ESTERIFICATION AND TRANSESTERIFICATION ASSISTED BY MICROWAVE OF CRUDE PALM OIL. HETEROGENEOUS CATALYSIS

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Abstract— The principal objective of this study was to obtain alkyl ester from crude palm oil (CPO), using microwaves like heating source, in a process of two stages by means of heterogeneous catalysis; the first stage (esterification), was made using Dowex 50X2, Amberlyst 15 and Amberlite IR-120 resins catalysts, to diminish the acid value of the oil, avoiding the soap formation and facilitating the separation of the phases. The second stage (transesterification) was made using potassium carbonate catalyst. The behavior in the crystallization of the product using differential scanning calorimetry, cloud point ASTM D2500 (2005) and pour point ASTM D97 (2005) was evaluated. The obtained biofuels fulfill the requirements of the American standards for biodiesel and the propose methodology for the synthesis presents environmental advantages and of increase in the reactivity, as opposed to the traditional methods of heating.

Keywords — Esterification, Transesterification, palm oil, microwave, heterogeneous catalysis.

I. INTRODUCTION

The conventional techniques of synthesis for the transesterification reaction with high yields commonly use homogeneous catalysts from inorganic acids and bases, preferentially, sodium and potassium hydroxides and alkoxides. The use of these catalysts have technical and environmental disadvantages, because of the neutralization processes of its salts, which in turn, generate cost overruns on the separation and purification of final product. In the last years, the works reported on development of heterogeneous catalysts for the transesterification reactions in acid and basic medium, have been quite extensive. (Kaita et al., 2002; Furuta et al., 2004; Lin and Radu, 2006; Bossaery, 1999; Park et al., 2008; Mbaraka, 2003; Bournay et al., 2005; Basu and Norris, 1996; Stern et al., 1999; Sanjib and Anju., 2005; Suppes et al., 2001; Barakos et al., 2007; Arzamendi et al., 2008; Kim et al., 2004; Xie et al., 2007; Gryglewicz, 1999; Bournay et al., 2005; Haitao and Xie, 2006; Kiss et al., 2006; Corma et al., 1998; Leclercq et al., 2001; Ma et al., 2008; Chen et al., 2007; D'Cruz et al., 2007), standing out clearly the trend of replacing these homogeneous catalysts, and this contributes to the elimination of additional stages of the process and improve the economy of the same. Because of its advantages, heterogeneous catalysts: they can be removed from of the reaction medium, can be recycled, the chemical structure and in some cases allows its holder guide reactions to certain products or reduce those that are undesirable, they offer the possibility of developing friendly processes with the environment, allow to be used for long periods of time without losing business and do not present dangers in handling or storage (Marchetti *et al.*, 2007).

Very few studies were performed to obtain alkylesters and all are obtained by homogeneous catalysis (Lee *et al.*, 1995). Yields of these reactions are very low by the high steric hindering that presenting the branched alcohols.

Microwaves are electromagnetic radiation with a frequency between 300 MHz to 300 GHz, where the most commonly used domestically and industrial, is that of 2450 MHz (Bougrin and Loupy, 2005). It was discovered in 1946 by Dr. Percy and only until 1980 was employed in different areas of chemistry.

Almost any type of organic reaction requiring heating or thermal conditions can be performed using microwave radiation. Microwave dielectric heating is dependent on the ability of a solvent or matrix to absorb microwave energy and to convert it into heat (Lidström *et al.*, 2001). The matrix absorbs the radiation by two mechanisms: dipole polarisation and conduction.

When irradiated at microwave frequencies, the ions or dipole of the sample align in the applied electric field. As the applied field oscillates, the dipole or ion field attempts to realign itself with the alternating electric field and, in the process, energy is lost in the form of heat through molecular friction and dielectric loss. The amount of heat generated by this process is directly related to the ability of the matrix to align itself with the frequency of the applied field. If the dipole does not have time to realign, or reorients too quickly with the applied field, no heating occurs (Perreux and Loupy, 2001)

Microwave irradiation produces efficient internal heating (in situ heating), resulting in even heating throughout the sample, as compared with the wall heat transfer that occurs when an oil bath is applied as an energy source. Consequently, the tendency for the initiation of boiling is reduced, and superheating above the boiling point of the solvent (Kappe, 2004)